

METHYL ETHYL KETONE

2500



MW: 72.11

CAS: 78-93-3

RTECS: EL6475000

METHOD: 2500, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA : 200 ppm
NIOSH: 200 ppm: STEL 300 ppm
ACGIH: 200 ppm: STEL 300 ppm
 (1 ppm = 2.95 mg/m³ @ NTP)

PROPERTIES: liquid; d 0.805 g/mL @ 20 °C;
 BP 79.6 °C; MP -86 °C; VP 13 kPa
 (100 mm Hg; 13% v/v) @ 25 °C;
 explosive range 2 to 10% v/v in air

SYNONYMS: 2-butanone; MEK

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (carbon molecular sieve, 160 mg/80 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	methyl ethyl ketone (MEK)
VOL-MIN:	0.25 L @ 200 ppm	DESORPTION:	1 mL CS ₂ ; stand 30 min
-MAX:	12 L	INJECTION VOLUME:	5 µL
SHIPMENT:	routine	TEMPERATURE-INJECTION:	250 °C
SAMPLE STABILITY:	6 weeks @ 25 °C [1]	-DETECTOR:	300 °C
BLANKS:	2 to 10 field blanks per set	-COLUMN:	55 to 75 °C
ACCURACY		CARRIER GAS:	N ₂ or He, 25 mL/min
RANGE STUDIED:	295 to 1180 mg/m ³	COLUMN:	glass or stainless steel, 4 m x 2-mm ID; 20% SP-2100/0.1% Carbowax 1500 on Supelcoport 100/120
BIAS:	6.21%	CALIBRATION:	MEK solutions in CS ₂
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.069 [1]	RANGE:	0.15 to 5 mg per sample
ACCURACY:	± 17.83%	ESTIMATED LOD:	0.004 mg per sample [1,2]
		PRECISION (\hat{S}_p):	0.04 [1]

APPLICABILITY: The working range is 17 to 560 ppm (50 to 1650 mg/m³) for a 3-L air sample. The method is applicable to 15-min samples. This method was developed to give improved sample stability compared to conventional charcoal tubes [2,3]. Side-by-side comparisons of this method and Method S3 were made in a sporting goods manufacturing plant in which MIBK, T HF, and toluene were also present. This method has also been used successfully for acetone [4].

INTERFERENCES: Under the given conditions, acetone and isopropanol have retention times similar to MEK. Mass spectrometry and other GC columns, e.g., SP-1000, or 30 m x 0.32-mm WCOT capillary coated with 1 µm DB-1, are aids to resolving interferences.

OTHER METHODS: This method is similar, except for the sampler, to Methods P&CAM 127 [5] and S3 [6,7], which it replaces.

REAGENTS:

1. Eluent: Carbon disulfide*, chromatographic quality, containing 0.1% (v/v) benzene* or other suitable internal standard.
2. Methyl ethyl ketone
3. Nitrogen or helium, purified.
4. Hydrogen prepurified.
5. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID. Two sections of 60/80 mesh carbon molecular sieve separated by 2-mm foam plug (front = 160 mg, back = 80 mg) (SKC Cat. No. 226-81, Supelco ORBO-90, or equivalent).
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 2500-1).
4. Vials, glass, 2-mL, PTFE-lined crimp caps.
5. Syringe, 10- μ L, readable to 0.1 μ L.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C). Work with it only in a hood. Benzene is a human carcinogen.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size of 0.25 to 12 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least five working standards over the range 0.004 to 5 mg MEK per sample.
 - a. Add known amounts of MEK to eluent in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (ratio of peak area of analyte to peak area of internal standard vs. mg MEK).
9. Determine desorption efficiency (DE) at least once for each batch of Amborsorb XE-347 used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount of MEK directly onto front sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. mg MEK recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 2500-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
NOTE: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area. Divide the peak area of analyte by the peak area of internal standard on the same chromatogram.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of MEK found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C, of MEK in the air volume sampled, V (L):

$$C = \frac{W_f + W_b - B_f - B_b}{V} \cdot 10^3, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

The method was evaluated with spiked samplers and with atmospheres generated by syringe pump/air dilution, verified by infrared absorption. Breakthrough (80% RH, 200 ppm, 0.3 L/min) = 16.4 L; DE (4 to 18 mg per sample) = 1.03; storage stability (0.7 to 4 mg per sample) = 90% after six weeks at 25 °C; precision and accuracy as given on page 2500-1 (15 samples) [1]. A user check gave an estimated LOD of 0.004 mg MEK per sample [2].

REFERENCES:

- [1] Slick, E. J., NIOSH, unpublished data (1983).
- [2] User check, UBTL, NIOSH Seq. #3990-J (unpublished, August 29, 1983).
- [3] User check, DataChem Labs., NIOSH Seq. #7053-L (unpublished, August 9, 1990).
- [4] DataChem Labs, NIOSH Seq. #7373-L (unpublished, Nov. 27, 1991).
- [5] NIOSH Manual of Analytical Methods, V. 1, P&CAM 127, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [6] Ibid., V. 2, S3, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [7] Documentation of the NIOSH Validation Tests, S3, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

METHOD REVISED BY:

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